

Abrasive Contaminants and Their
Effect on Coating Performance

Task

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January 1990

By

John Peart

Report Documentation Page				Form Approved OMB No. 0704-0188	
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1. REPORT DATE JAN 1990		2. REPORT TYPE N/A		3. DATES COVERED -	
4. TITLE AND SUBTITLE Abrasive Contaminants and Their Effect on Coating Performance				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Surface Warfare Center CD Code 2230 - Design Integration Tools Bldg 192 Room 128 9500 MacArthur Blvd Bethesda, MD 20817-5700				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release, distribution unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	18. NUMBER OF PAGES 32	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

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A B S T R A C T

Previous National Shipbuilding Painting Research demonstrated that substrate residues from salt contaminated slag abrasives cause rust-back and a possible reduction of coating life.

This study characterizes eight (8) commercially available abrasive products for the water lechate conductivity, chloride and sulfate content and their effect on rust-back and coating performance in ambient and pressurized deionized water immersion.

Four copper slag products were examined petrographically to identify the minerals in the slag particles and to determine if free copper was present.

Scanning Electron Microscopy - Energy Dispersive X-Ray Fluorescence (EDXRF) analysis was used to identify slag constituents.

ACKNOWLEDGEMENT

This research project was produced for the National Shipbuilding Research Program as a cooperative cost-shared effort of the Maritime Administration, the U.S. Navy, and National Steel and Shipbuilding Company (NASSCO), The Surface Preparation and Coatings Panel SP-3 of the Society of Naval Architects and Marine Engineer's (SNAME) Ship Production Committee sponsored the project.

Program definition and guidance was provided by members of the SP-3 committee of SNAME and Mr. James Ruecker/Mr. Lyn Haumschilt, NASSCO program managers.

This study was conducted and its report prepared by John Peart. Benjamin Fultz provided valuable assistance in organizing test panel preparation and providing technical commentary on the final report.

Special thanks is given to Dr. Wolfgang Baum of Pittsburgh Mineral and Environmental Technology (PMET) who performed the petrographic and scanning electron microscopy (SEM) studies and the energy dispersion x-ray analysis and also Dr. Bernard Appleman and Dr. Simon Boocock of Steel Structures Painting Council (SSPC) who performed the high pressure water immersion test and laboratory soluble ion analysis.

The special support and assistance offered by the following people and companies is gratefully acknowledged.

Mr. William Hitzrot	Chesapeake Specialty Products
Mr. David Bloodgood	Devoe Coating Co.
Mr. Bud Hedrick, II	Apache Abrasives
Mr. Larry Bolander	Kleenblast
Mr. David Dorrow	Reed Minerals
Mr. Bryan Rabon	Custom Blast Inc. Brock Industries

EXECUTIVE SUMMARY

The shipbuilder as well as the ship owner is concerned with the quality of the ship's painting and wants insurance that the coating system will perform effectively to control corrosion and steel loss for its predicted life. The quality and cleanliness of the abrasive used in the surface preparation process has a significant impact on the coating system performance; therefore, abrasive procurement specifications must reflect objective requirements that characterize available quality products that will produce the required substrate for a quality coating application. Secondly, the shipyard requires not only a quality abrasive product, but also competitive sources of supply.

If unrealistic material specification requirements are promulgated based on inadequate data or conjecture, some competitive sources are eliminated and ship surface preparation and painting costs escalate significantly. In response to this issue, this study characterizes eight commercially available copper and coal slag abrasives for water soluble contaminants capable of being transferred to a clean substrate and evaluated their effect on coating performance. It was determined that the eight products tested produced acceptable substrates for the application of coatings for immersion service.

Petrographic examinations, scanning electron microscope and energy dispersive x-ray analysis were performed on three copper slag abrasive products marketed in the United States. Copper compounds were identified in all three. Two of the products were included in the pressurized and ambient deionized water

immersion testing phase of the study. Test results determined no ill effects on coating performance.

The study results did not support the rust-back and blistering phenomenon reported by the Navy Mare Island Paint Laboratory investigation⁽³⁾. Nor did it confirm the theory concerning rust-back due to the development of free copper cathodic sites on the substrate. The findings of the referenced work were the primary data on which the Navy based many of the requirements of specification Mil-A-2226A(SH) for abrasives.

It is recommended that the reader should, as a minimum, read the conclusion of this report. Additionally, the test results and discussion should be of particular interest to those involved in abrasive specification development, procurement and material quality control functions.

1.0 Background

Abrasives processed from slags have replaced silica sands in many shipyards and steel fabrication shops because of their low free silica content. Copper smelters and coal fired electric generating plants are the major material sources for these slag products.

The chemical characteristics of the copper slags are dependent upon the mineral content of the ore feed, the completeness of the reduction reaction, processing parameters and their exposure to contaminants during and after air or water quenching.

The chemical make-up of furnace bottom slags is primarily dependent on trace elements in the coal source while the soluble salt content is primarily the result of contamination during quenching and storage.

Ore sources which may be predominantly mined for the recovery of one metal may often contain significant amounts of other metals which are commercially recoverable, e.g., lead, zinc, copper, arsenic, etc., as well as trace compounds of other metallics. Hence, smelter slags may contain unreduced minerals or metals tied up as compounds if the reduction reaction is incomplete in the smelting process.

Copper slags can be either air or water quenched (see Figures 1, 2). Bottom or coal slags are most often water quenched. Therefore, the purity of the quenching water and the cleanliness of the quenching basins have a significant effect on the cleanliness of the abrasive product.

Secondary copper smelters are also a source of copper slag. These smelters recover copper from scrap copper alloy products, e.g., telephone switching gear, electronic hardware, wrought products, wire, etc. Such slag may contain alloying elements, e.g., beryllium, iron, etc. The former being toxic and the latter easily oxidizable to rust which appears as spots on the abrasive cleaned steel surface.

The bottom or coal slags being products of an oxidation reaction, will concentrate trace metallic compounds associated with the coal source. Some of these trace elements may be radioactive resulting in a slag with a significant gamma activity.

OSHA requirements do not specify maximum acceptable limits of trace heavy metals in abrasives, but do establish maximum safe levels in the workers blood.

Abrasive EP-TOX test- leachates must have trace heavy metal levels below the hazardous classification requirement for disposal in landfills. Additionally, one state, California imposes strict total maximum heavy metal content requirements for a nonhazardous landfill disposal classification. Some abrasives may have difficulty meeting these California nonhazardous waste classification requirements. The use of a non-compliant abrasive would result in a hazardous classification and high disposal costs for the generator of the abrasive-paint debris.

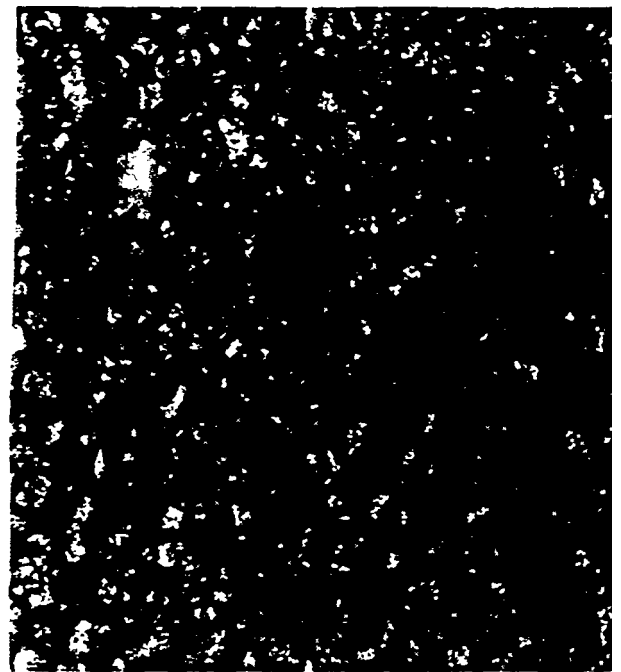
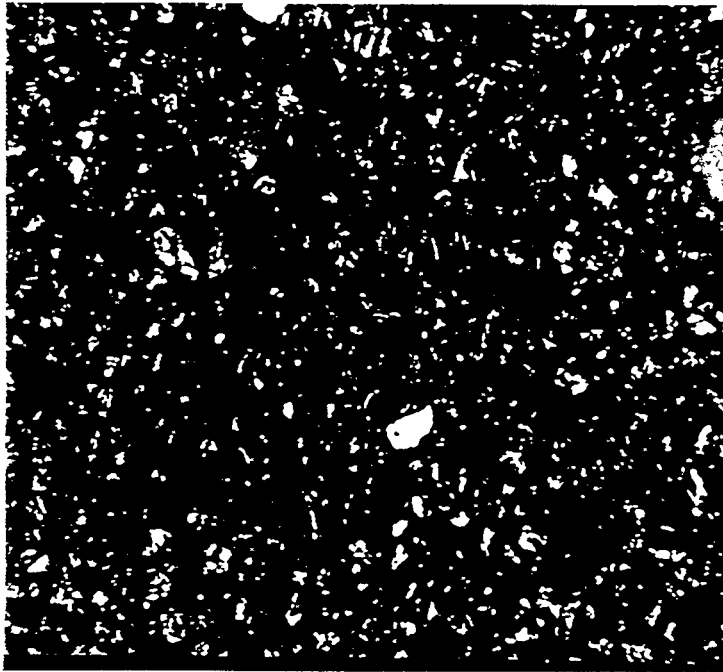


Figure 1 - Copper Slag Water Quenched

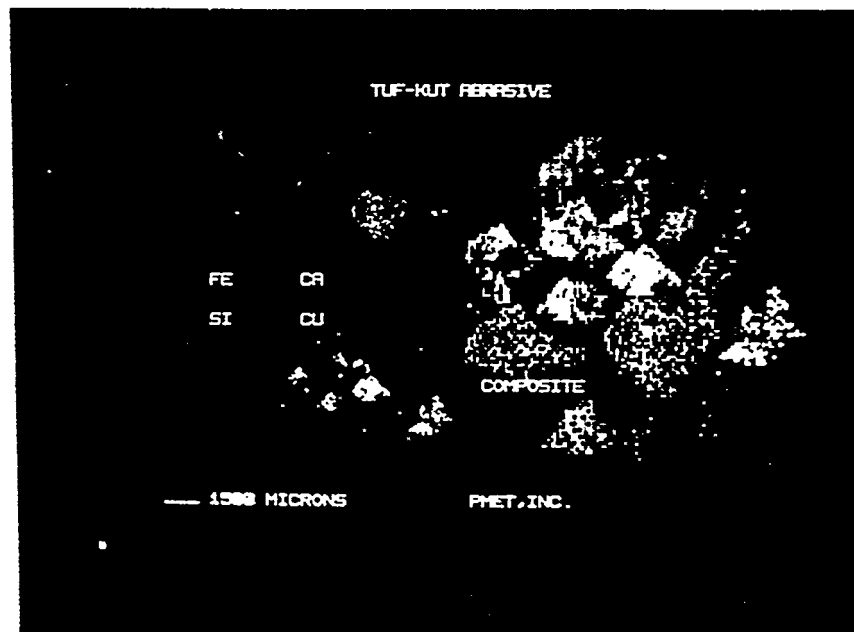


Figure 3- Japanese Copper Slag SEM-Energy Dispersive X-Ray Analysis

As stated previously, another major source of slag abrasive contamination is the purity of the quench water and the cleanliness of the holding basin. Contamination from these sources are more prevalent with coal slag abrasives. Coal fired power generating plants may use their slag quenching basins for receptors of other materials, e.g., fly ash, solid stack wastes, and scrubber residues mechanical mixing may occur in the quenching pond. If the slag is not adequately washed before crushing, contamination of the abrasive will result. These types of contaminants do not combine chemically with the slag but retain their own physical form in the mixture. These contaminants are often visible and appear on the abrasive cleaned substrate as small colored specks depending upon their source.

Soluble salt contaminants are most often of greater concern to the abrasive user because of their greater potential for reducing coating life and being unseen, often go undetected. Coal slags from power generating plants located in tidewater areas, which may use high salt content water for quenching because of its availability, are suspect. The salt content of the abrasive, from such slag source, should be determined by analysis of their water leachate.

The critical level of residual soluble salts on the substrate that may reduce coating life and the role of abrasives in their deposition is of great interest to those involved in protecting structures from corrosion. The National Shipbuilding and the Federal Highway Administration research programs both have contract studies in progress to enhance our limited knowledge in this area. The results are being awaited.

2.0 Introduction

Soluble salt residues on substrates to be painted have been observed to initiate and accelerate the corrosion of the substrate and to reduce coating performance.

W. C. Johnson⁽¹⁾ found that residual substrate chloride and sulfate salts on steel surfaces strongly attract and accelerate water transmission through the paint film, due to osmotic pressure, resulting in premature coating failure. The reference also discusses how salts cause deposition of moisture on the surface of the steel before paint application even though substrate temperatures are above the dew point. This condensation results from the lowering of water vapor pressure by the salt residue at the air-substrate interface.

Current paint specifications require that the dew point of the surface to be painted must be as a minimum 5°F above that of the dew point of the ambient air to prevent moisture condensation. If sodium chloride is present a 11 °F rather than 5°F differential is required to prevent condensation because of the resultant vapor pressure depression. This results in flash rusting of the steel surface. Other soluble salts decrease the dew point temperature similarly. The magnitude of the depression is dependent upon the chemical species present.

The result of this phenomenon was noted and documented by Mr. B. S. Fultz⁽²⁾ in his evaluation of citric acid cleaning of steel for the National Shipbuilding Research Program. He observed that abrasive blasted panels cleaned with a particular coal slag abrasive quickly oxidized and discolored at ambient laboratory conditions (75 degrees Fahrenheit and 55 percent relative humidity) while other panels cleaned concurrently with other abrasives did not. His subsequent investigation, as to the cause, determined that the problem abrasive contained high levels of sodium chloride. The salt contamination was traced to the coal slag source; a power generation facility that quenched the slag in brackish water.

Rapid evaluation of the degree of water soluble ion contamination of abrasives can be easily accomplished by measuring the conductivity of their water leachate. American Society of Testing Materials (ASTM) has developed a standard test method ASTM D4940 to determine this. The analysis is performed on the leachate from equal volumes of abrasive and reagent grade water. This specification standardizes the method of analysis but it does not propose an acceptable level of soluble salts for abrasives. A method note is included that defines 500 micromhos/cm (microsiemens) and 50 micromhos/cm (microsiemens) conductance measurements as high and low soluble salt containing abrasives respectively. Mr. W. C. Johnson⁽¹⁾ proposes categories of concentration ranges based upon leachate conductance testing of commercially available abrasives as follows:

High concentration 2,000 - 3,500 ohm/cm (500-286 microsiemens)

Medium concentration 7,100 - 8,300 ohm-cm (141-120 microsiemens)

Low concentration 15,000 - 39,000 ohm-cm (67-36 microsiemens)

The U.S. Navy, in Military Specification, MIL-A-22262A (SH) has established a maximum chloride content of 0.03 percent by weight when tested per ASTM D 1411, "Water-Soluble Chlorides Present as Admixes in Graded Aggregate Road Mixes." This specification also establishes a conductivity of less than 100 micro-mhos/cm (microsiemens) when measured in accordance with ASTM D1125, "Electrical Conductivity and Resistivity of Water." As opposed to the ASTM standard, 20 grams of abrasive is mixed with 500 ml of water. Using a specific gravity of 2.5, this roughly equates to a one part abrasive to sixty parts water by volume. Maximum metal content is also addressed with copper being of special interest because of tests performed at the Port Hueneme Laboratory and published as Paint Laboratory Technical Memorandum No. 134.6-9, "Evaluation of Abrasive Blast Materials, Chemical and Physical Test Results of Sampled Abrasive," dated September 30, 1984. Copper was reported as possibly causing rapid blistering in water immersion due to the initiation of galvanic cells on the substrate. This report also served as the basis for many other requirements for specification MIL-A-22262A (SH).

The new proposed Steel Structures Painting Council, (SSPC) "Mineral and Slag Abrasive" specification now under development does not address chloride or other specific ionic salts but does establish a conductivity limit of 200 micromho/cm microsiemens when tested in accordance with ASTM Standard D-1125. In this method, 100 grams of abrasive is added to 1500 ml of water, which equates to approximately forty parts water to one part abrasive.

Listed below is a comparison of ASTM, U.S. Navy, SSPC and Mr. Johnson's proposed limits of conductivity reported in microsiemen normalized to represent leachate from equal volumes of abrasive and water.

<u>ASTM Specification</u>	<u>MIL-A-22262A(SH)</u>	<u>SSPC</u>	<u>W. C. Johnson</u>
50 (Low)	6,000 (max)	8,000(max)	26-67 (Low)
500 (High)			120-140 (Medium)
			290-500 (High)

As can be seen from this variation of the proposed requirements, no uniformly accepted standard exists. It should also be noted that all the above limits were arbitrarily established and are not based on controlled testing to establish critical limits, i.e., the critical ion concentration at which premature coatings failure occurs.

The purpose of this study was to obtain commercially available abrasives, primarily of the slag types, measure conductance, identify specific ion contamination and to coat test panels prepared with these abrasives and expose them to an immersion test environment in an attempt to establish contamination which reduce coating performance.

3.0 Objectives

The objectives of the study were three fold:

- o The determination of the ion concentration of water leachates from representative abrasive blast media supplied to the U.S. Shipbuilding industry.
- o Determine if any of the evaluated representative abrasives cause rapid rust-back (flash rusting).
- o Determine if any of the evaluated abrasives cause blistering of paint films in immersion service.

4.0 Coating Test Design

Six copper slag and two coal slag abrasive products from six different suppliers were selected for evaluation. A G-40 steel grit was used as control for comparison purposes.

More copper slag abrasives were chosen primarily in an attempt to confirm the validity of the theory proposed by the Navy Laboratory investigators⁽³⁾ to **explain the** coating blistering experienced in their testing. The supposition was that the problem copper slags deposited free cathodic copper sites on the substrate which developed anodic sites on the steel that initiated galvanic corrosion and subsequent coating blistering.

Triplicate six inch (6") by six inch (6") panels were blasted to SSPC-SP-5 White metal using each test abrasive and the G-40 steel grit control for a total of twenty-seven panels. All the panels were cut from the same piece of new, 1/4 inch thick, A-36 steel with intact mill scale. Two panels from each set were coated with the VOC compliant version of the U.S. Navy Formula 150/151 epoxy coating system. The resultant dry film thickness of the two coat systems ranged from 14 to 16 mils. See Table 1 for a list of abrasives tested, profile and actual dry film thicknesses of each panel set as applicable.

Samples of each abrasive product in the 'as received' condition were collected and retained for the determination of pH, conductance and soluble salt concentrations of the leachate. Conductance and pH were measured in the laboratory, using laboratory equipment and in a field environment, using hand held, pocket instruments. The pH was also measured using a "Chemetrics Vacuette" calorimetric field test.

Leachable chloride concentrations were measured using both field and laboratory equipment. Leachate sulfate concentration was determined by laboratory analysis only.

After being abrasive blast cleaned, the uncoated panels were placed in a controlled environment and observed for rust-back during a period of seven days. Pictures were taken at regular intervals. Ambient relative humidity varied from 55 to 70 percent and temperature from 70 to 75 degrees F.

Coated panels sets were placed in two different immersion test environment. One set was tested in distilled (DI) water in a pressure chamber at 100 degrees F for 1000 hours. The pressure of the chamber was maintained between 52 - 55 psig.

In the second test, coated panels were immersed in DI water for six months. The DI water was at ambient temperature condition for the first week of exposure. It was then changed and the replacement water was maintained at 82 degrees F for thirty days. The DI water was again replaced and for the second thirty days of immersion the DI water cycled at 82 degrees F for five days, then at 120 degrees F for eight hours for two days. This resulted in 64 hours immersion at 120 degrees F.

For the next four months, the DI water was maintained at ambient temperature which ranged from 76 degrees to 90 degrees F.

Table 1. Abrasive Sources and Test Panel Data

No.	Abrasive	Panel Nos.	Profile	Dry Film Thickness
1	Chesapeake Specialty Products Metgram G-40 Steel Grit	1, 2, 3	1.9	15 mils
2	Tidewater Marine Sure Shot 16-30 Coal Slag	4, 5, 5	2.9	16 mils
3	Apache Tennessee 30-50 Copper Slag	7, 8, 9	2.5	16 mils
4	Apache El Paso 20-50 Copper Slag Quenched	10, 11, 12	3.1	15 mils
5	Apache El Paso 20-50 Copper Slag Ambient Cooled	13, 14, 15	3.4	16 mils
6	H. B. Reed Black Beauty 1240 Coal Slag	16, 17, 18	3.3	14 mils
7	Kleenblast All Purpose #1030 Copper Slag	19, 20, 21	3.7	14 mils
8	Rocky Mountain Energy All Purpose Copper Slag	22, 23, 24	2.8	15 mils
9	Abrasifs Mendiola Bayonne France Copper Slag	25, 26, 27	3.1	15 mils
10	Clean-Blast Tacoma NA II TruGrit 16-3	Petrographic/scanning electron microscope Examination and EDXRF analysis		
11	Barnes Japanese Tuf-Kut 8-12	Petrographic/scanning electron microscope Examination and EDXRF analysis		
12	Apache 30-50 Copper Hill, TN	Petrographic/scanning electron microscope Examination and EDXRF analysis		
13	Apache 10-50	Petrographic/scanning electron microscope Examination and EDXRF analysis		

5.0 Leachate Analysis

All analysis were performed on a water leachate prepared using equal volumes of test abrasive and reagent grade water.

5.1 Laboratory

5.1.1 The laboratory pH measurement was determined using standard pH meters.

5.1.2 The sulfate concentration was determined in accordance with APHA Method 426C of Standard Methods for the Analysis of Water and Waste Water, 16th Edition. The prepared sample was filtered and the turbidity was measured after the addition of barium chloride. A visible spectrometer with a detection limit of approximately 1 part per million (ppm) was used to measure the degree of turbidity.

5.1.3 The chloride concentration of the leachate was determined by using three different techniques. The first was the APHA Standard Method 407B (1980) which uses a "Titret." The second and third methods used the following ion selection electrodes:

° Orion Research, Inc.

Meter 94-17 chloride electrode and 96-17B

Combination chloride electrode instruction manual

^oFisher Scientific, Inc.

Silver-Silver Chloride Reference Electrode and
Chloride Sensing Ion-Selective Electrode

5.1.4 Conductance was measured using standard laboratory equipment and procedures.

5.2 Field

5.2.1 The pH of two samples were measured using a "Vacuettes" pH kit, model K-7738. The K-7738 kit employs a stable, liquid indicator which gives accurate pH measurements. The accuracy of this test kit is not affected by variations in the ionic strength, sample matrix or temperature. Since chloride in concentration of 1 ppm or more could interfere with the test, a drop of neutralizer is added to the prepared sample. Twenty-five milliliters (ml) of each sample was measured into a container. The appropriate "Vacuette", depending on expected pH, was selected and the tip inserted into the same. The tip of the inserted "Vacuette" was broken and the sample was drawn into the vacuum tube. The prepared sample and indicators in the vacuette were mixed and then visually compared to a set of colored liquid standards. The reference standard that matched the color of the sample determined its pH.

A hand held, Cole-Parmer Pocket pH Meter was the second method used to determine the pH of the leachate samples.

In one case the pH as measured using the "Vacuette" kit was measured 4.8; whereas, the hand held instrument read 4.7.

In another sample set the "Vacuette" technique determined a pH greater than 8. The pH meter reading for the same sample was 7.9. The hand held meter was standardized using a known buffered solution.

From the comparison of field and laboratory results and the accuracy and precision of the instrument measurements of buffered solutions of known pH, it was determined that the hand held, pocket pH meter was an excellent tool for field measurements of pH.

5.2.2 A Chemetrics, hand held, "Titrets" titration cell kit, model K-2050 (20 to 200 ppm) and model K-2051 (200 to 2,000 ppm) were used to measure chloride concentrations. The leachate sample was filtered prior to the analysis.

The "Titret" test kit is based on miniature titrimetric chemistry which used mercuric nitrate in an acidic solution to react with any chloride present in the sample. Since the kit employs a reverse titration technique, all common interferences associated with standard visual titrations no longer contribute to the determination error.

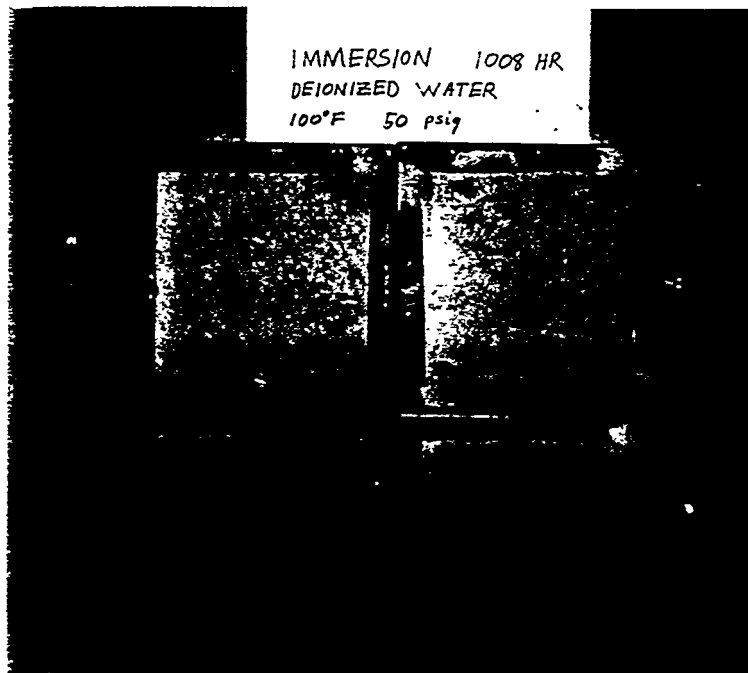
Fifteen mls of sample were added to the container supplied with the kit. Activator solution was then added to the sample. The sample was titrated using the glass vacuum ampules and "Titrettor" supplied with the kit. When the color in the "Titrettor" turned from purple to light yellow, the titration was complete and the results were then read directly from graduations on the ampule.

5.2.3 Field measurement of conductance was made using a Cole-Parmer Model 5941-00 hand held, pocket meter with a range of 10 to 1990 microsiemens. The instrument was standardized using standard solutions of 84 or 1413 microsiemens depending upon the conductance range of the sample being tested.

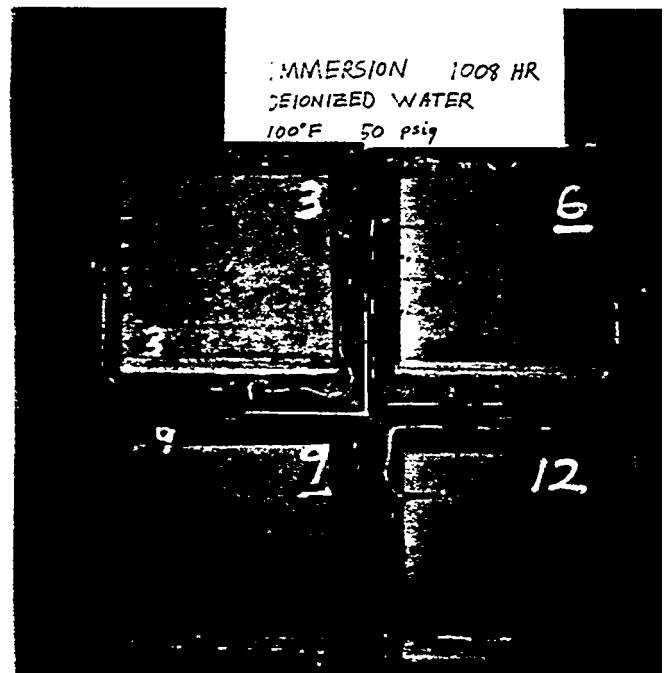
6.0 Immersion and Analytical Test Results

6.1 Immersion

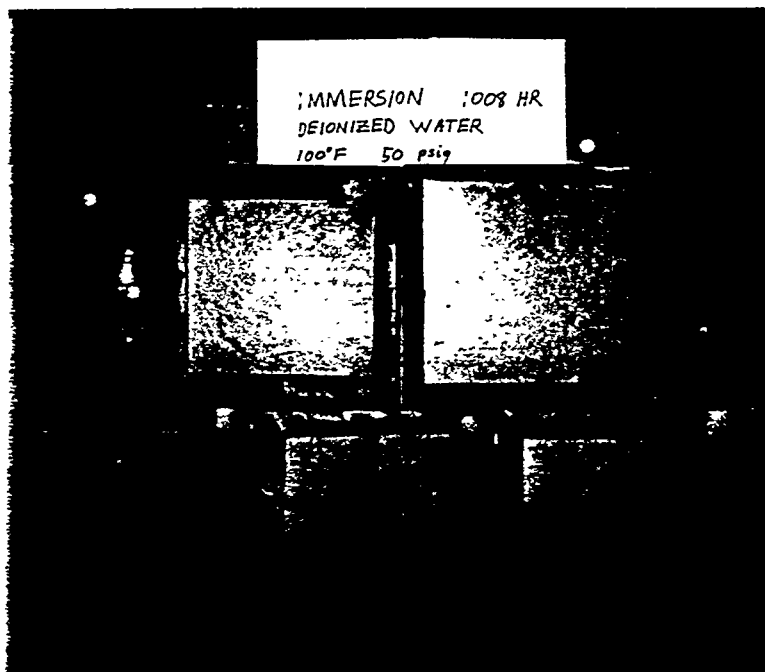
None of the painted surfaces prepared by the test abrasives showed rusting or blistering in either the ambient or 50 psig pressured distilled water immersion tests. Hence, it is concluded that the abrasive products tested are suitable for use in preparing surfaces for painting in immersion applications (see Figure 4).



OBVERSE



REVERSE



OBVERSE



REVERSE

Figure 4. immersion DI water. 1008 hrs @ 100 °F 50 PSIG
Panel /Sample Nos. - Abrasive Nos. in Parentheses ()
3(1), 5(2), 9(3), 12(4), 15(5), 18(6), 21(7), 24(8), 27(9)

Both the ambient pressure deionized water at 120 °F and the pressurized deionized water tests are harsh in nature and are effective indicators of film integrity and the presence of any contaminants that may induce osmotic pressures and accelerated moisture transfer through the paint film.

New hot rolled-steel panels with intact mill scale were used to insure that if any significant salt contamination occurred it was the result of an abrasive transfer.

The contaminants associated with the tested abrasives were not of sufficient quantities and/or were not transferred to the clean substrate to a degree as to cause a reduction in coating performance.

6.2 Leachate Analysis

Table 2 contains a compilation of the laboratory and field measurements of sulfate, chloride levels and leachate conductivity. Units of conductance are listed in microsiemens.

Table 2. Leachate Analysis

Abrasive No.	p H		Chl ori de		Parts/Mill ion		Sul phate Parts/ Mill ion Lab	Conductance Mi crosi emens	
	Fi el d	Lab	Fi el d / Ti ter	Lab Ti ter	Lab No. 1	Electrode No. 2		Fi el d /	Lab
1	4.7	5.2	<20	6.0	2.69	3.72	62	165	140
2	5.7	5.9	<20	2.1	1.21	2.10	255	460	540
3	9.7	11.2	<20	4.9	3.24	6.30	102	445	520
4	6.7	8.1	<20	2.0	1.04	1.78	4	20	40
5	6.5	7.5	<20	2.2	1.41	2.20	55	210	200
6	6.5	8.3	<20	NA	0.71	0.75	0	20	20
7	6.2	8.0	<20	9.0	9.82	11.4	129	280	180
8	6.4	7.0	<20	18	21.10	21.10	129	360	340

Both the laboratory and field techniques for measuring chloride with the "Titret" use the same type of chemicals except that the laboratory kit is more sensitive.

Comparison of the different laboratory techniques demonstrated good correlation of results, between the specific electrode ion electrode, and the "Titret" kits.

Since the "Titret" is suitable for field use, it is the recommended method.

The pH results using field and laboratory methods were similar in magnitude but differed by as much as 1.8 pH units. This variance may have resulted from a difference in the pH of the reagent water used. The commercially available water for field use has a suitable low electrical resistance for conductance testing but generally has a low pH value due to the absorption of carbon dioxide from the air. The pH of the DI water available in the field ranged from 5.6 to 5.8 as compared to a desired neutral pH reading of 7.0. This may be the reason that in all cases, the field pH determinations were lower than the laboratory measurements.

6.3 Mineralogical Examination

The results of the mineralogical examination and the SEM-EDXRF analysis are summarized below.

Sample No. 10	No metallic copper observed. SEM work shows trace of copper in a few slag particles. This copper may, however, be associated with copper-bearing sulfides and/or secondary copper minerals which formed after the slag production.
---------------	--

- Sample No. 11 This sample contains noticeable amounts of metallic copper (see Figure 3). In addition, there are secondary copper minerals (probably malachite). The metallic copper is alloyed with approximately 10% iron (semi-quantitative SEM-EDXRF analysis).
- Sample No. 12 No metallic copper observed. SEM-EDXRF work shows trace of combined copper in a few slag particles.
- Sample No. 13 The slag particles of this sample appear to contain appreciable amounts (>2%) of residual sulfides from the ore, some of which was copper bearing. Metallic copper was not detected.

The Japanese product (sample 11) Figure 3 was the only abrasive examined that contained notable amounts of metallic copper. It was alloyed with approximately 10% iron. In addition, there were secondary mineral present, most likely malachite. The material is no longer being marketed in the United States and was not available to be included in the performance testing or leachate analysis.

The slag source from which the abrasive was produced could not be identified.

The other Kleen-Blast and Apache products examined contained no free copper but trace amounts of copper were present in a combined form. These products did not cause rust back or reduced coating performance as documented by the test results obtained in this study .

7.0 Conclusions

The abrasives tested did not produce flash rusting or reduce the ambient of the pressurized deionized water immersion performance of the Navy Formula 150/151 epoxy coating system. Expressed conversely, the abrasives tested were found to be acceptable for use in preparing surfaces for coating in immersion service applications.

Equal volume abrasive/water leachate conductance levels in the 0 - 550 microsiemen range did not adversely affect the performance of the coating system tested.

The exposure environment history and the degree of contamination of the surfaces being blasted appears to have a more critical impact on the concentration of residual salt on the cleaned substrate than the concentration of ionic constituents in the process abrasive.

Field instrument measurements of abrasive leachate conductance correlate well with more sophisticated laboratory instrumentation. Their use in conjunction with ASTM D4940 provide a fast, accurate and reproducible method to determine the degree of ionic contamination of the abrasive product being used.

"Titret" kits of the appropriate concentration range provide reliable field determination of chloride concentrations of abrasive/water leachate.

A good sulfate field test method of appropriate sensitivity has not been identified.

Metallic copper was identified in only one of the copper slags examined. The material is no longer marketed in the United States. Sufficient material was not available to determine its possible effect on coating performance. The other copper slag abrasives examined contained traces of combined copper; however, subsequent accelerated corrosion testing did not indicate any resultant ill effect on coating performance. Study test results did not duplicate the rust-back and blistering experienced by the Navy investigators⁽¹⁾ even though some of the abrasive material sources was identical. The reference was not specific as to the 150/151 product used in the test. The Rule 66 version which may have been the material tested, has demonstrated a tendency to blister in immersion due to the partial water solubility of one of the solvent system components.

No correlation between copper content and rust-back or blistering was observed, nor did the test results substantiate the copper disposition-anodic site initiation theory of rust-back postulated by reference (3).

8.0 Bibliography

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